

PATENT ABSTRACTS OF JAPAN

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(71)Applicant : SUMITOMO CHEM CO LTD

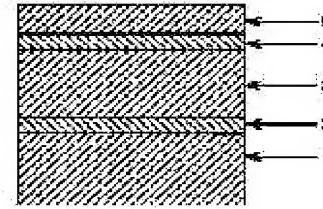
(72)Inventor : IECHIKA YASUSHI
TAKADA TOMOYUKI
ONO YOSHINOBU

(54) FABRICATION OF III-V COMPOUND SEMICONDUCTOR

(57)Abstract:

PROBLEM TO BE SOLVED: To form light emitting elements exhibiting uniform emitting state with high yield by forming a first layer represented by a specified formula and then feeding not a group III material but a carrier gas and/or a group V material prior to growth of a second layer.

SOLUTION: A buffer layer 2 is grown on a substrate 1 followed by growth of a Ga_xAl_{1-x}N layer 3, a first layer 4 of III-V compound semiconductor represented by formula I, and a second layer 5 represented by formula II. The layer 4 becomes a quantum well layer by employing a double heterostructure where the band gap of the layers 3, 5 is larger than that of the layer 4 and intensive photoluminescence is generated from the quantum well layer. When a III-V compound semiconductor is employed in a light emitting element, the light emitting elements can be formed with uniform wavelength and intensity in the surface of substrate. The effect is especially conspicuous in a layer containing InN more than the mixed crystal ratio, i.e., 5%.

In_xGa_{1-x}N (0 < x < 1),
0 < y < 1, 0 < z < 1Ga_xAl_{1-x}N (0 < x < 1),
0 < y < 1, 0 < z < 1

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CLAIMS

[Claim(s)]

[Claim 1]Vapor phase epitaxy of the 1st layer of a 3–5 fellows compound semiconductor expressed with general formula $In_xGa_yaluminum_zN$ (however, $x+y+z=1$, $0 < x \leq 1$, $0 \leq y \leq 1$, $0 \leq z \leq 1$) is carried out, Next, in a method of carrying out vapor phase epitaxy of the 2nd layer expressed with general formula $In_uGa_valuminum_wN$ (however, $u+v+w=1$, $0 \leq u \leq 1$, $0 \leq v \leq 1$, $0 \leq w \leq 1$), and manufacturing a 3–5 fellows compound semiconductor, A manufacturing method of a 3–5 fellows compound semiconductor having the process of supplying carrier gas or supplying carrier gas and five group materials without supplying three group materials before growth of the 2nd layer, after growing up the 1st layer.

[Claim 2]A manufacturing method of the 3–5 fellows compound semiconductor according to claim 1, wherein a mixed crystal ratio of InN of the 1st layer is not less than 5%.

[Claim 3]A manufacturing method of the 3–5 fellows compound semiconductor according to claim 1 or 2 with which thickness of the 1st layer is characterized by not less than 10A being 500A or less.

[Claim 4]A manufacturing method of the 3–5 fellows compound semiconductor according to claim 1, 2, or 3, wherein each concentration of each element of Si, germanium, Cd and Zn which are contained in the 1st layer, and Mg is below $10^{19} cm^{-3}$.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention]This invention relates to the manufacturing method of a 3–5 fellows compound semiconductor, especially the manufacturing method of the 3–5 fellows compound semiconductor for light emitting devices.

[0002]

[Description of the Prior Art]Conventionally as a material of light emitting devices, such as a laser diode of the light emitting diode (it may be hereafter described as LED.) of ultraviolet, blue, and green regions or ultraviolet, blue, and green regions, The 3–5 fellows compound semiconductor expressed with general formula $In_xGa_yaluminum_zN$ (however, $x+y+z=1$, $0 < x \leq 1$, $0 \leq y \leq 1$, $0 \leq z \leq 1$) is known.

[0003]Since a crystal with this compound semiconductor good in bulk growth is not obtained, homoepitaxial growth using this compound semiconductor itself as a substrate is difficult. By the way, it is known that a good crystalline thing can be obtained because the compound semiconductor expressed with general formula $Ga_aaluminum_bN$ (however, $a+b=1$, $0 \leq a \leq 1$, $0 \leq b \leq 1$) uses buffer layers, such as GaN and AlN. This

$Ga_aaluminum_bN$ has the five same group elements as this $In_xGa_yaluminum_zN$, and since a crystal structure is also the same, this $In_xGa_yaluminum_zN$ can obtain a high crystalline thing by growing up on the above-mentioned $Ga_aaluminum_bN$.

[0004]By the way, the grating constant of this compound semiconductor changes with mixed crystal ratios a lot. Especially, to GaN or AlN, about 12% or more, since it is large, depending on the mixed crystal ratio of each class of this compound semiconductor, a big difference may produce the grating constant of InN in the grating constant between layers. When there is big lattice mismatching, a defect may arise into a crystal and it becomes the cause of reducing crystallinity. It is difficult to realize high luminous efficiency in the light emitting device which generally produced the defect using the crystal included mostly.

[0005]In order to suppress generating of the defect by lattice mismatching, according to the size of distortion by lattice mismatching, layer thickness must be made small. However, when thickness made a film an active layer dramatically, it was difficult for the physical properties of a luminous layer to receive influence also in the mixed crystal ratio of a luminous layer or the deflection of some of thickness, and to produce the target luminous wavelength or the light emitting device of luminescence intensity uniformly covering all the substrates faces.

[0006]

[Problem(s) to be Solved by the Invention]It is uniform, the purpose of this invention has a uniform luminescent state, when it uses as the manufacturing method of the 3–5 fellows compound semiconductor which can manufacture the high 3–5 fellows compound semiconductor thin film of the yield, especially a light emitting device, and there is in providing the manufacturing method of the 3–5 fellows compound semiconductor for light emitting devices which can obtain the high light emitting device of the yield.

[0007]

[Means for Solving the Problem]This invention persons found out that a uniform thin film was obtained for high quality by Lycium chinense in a process of stopping supply of three group materials as a result of wholeheartedly examination by the time it grows up the following layer after growing up a thin film of this compound semiconductor about a growing condition of a thin film of this 3–5 fellows compound semiconductor, and resulted in this invention.

[0008]That is, this invention is an invention described below.

[1]Vapor phase epitaxy of the 1st layer of a 3–5 fellows compound semiconductor expressed with general formula $In_xGa_yaluminum_zN$ (however, $x+y+z=1$, $0 < x \leq 1$, $0 \leq y \leq 1$, $0 \leq z \leq 1$) is carried out, Next, in a method of

carrying out vapor phase epitaxy of the 2nd layer expressed with general formula $In_u Ga_v aluminum_w N$ (however, $u+v+w=1$, $0 \leq u \leq 1$, $0 \leq v \leq 1$, $0 \leq w \leq 1$), and manufacturing a 3–5 fellows compound semiconductor, A manufacturing method of a 3–5 fellows compound semiconductor having the process of supplying carrier gas or supplying carrier gas and five group materials without supplying three group materials before growth of the 2nd layer, after growing up the 1st layer.

[2]It is characterized by a mixed crystal ratio of InN of the 1st layer being not less than 5%. [1]A manufacturing method of a 3–5 fellows compound semiconductor of a statement.

[0009][3]Thickness of the 1st layer is characterized by not less than 10A being 500A or less. [1]or[2]A manufacturing method of a 3–5 fellows compound semiconductor of a statement.

[4]Each concentration of each element of Si, germanium, Cd and Zn which are contained in the 1st layer, and Mg is characterized by being below $10^{19} cm^{-3}$. [1]**[2]or[3]A manufacturing method of a 3–5 fellows compound semiconductor of a statement.

[0010]

[Embodiment of the Invention]Next, this invention is explained in detail. Generally as a substrate for crystal growth of this 3–5 fellows compound semiconductor, sapphire, ZnO, GaAs, Si, SiC, NGO ($NdGaO_3$), a spinel ($MgAl_2O_4$), etc. are used. Since the quality crystal of a large area is obtained transparently, especially sapphire is preferred.

[0011]as the manufacturing method of this 3–5 fellows compound semiconductor — molecular beam epitaxy (it may be hereafter described as MBE.) — law and organic metal vapor growth (it may be hereafter described as MOVPE.) — law and hydride vapor phase epitaxy (it may be hereafter described as HVPE.) — law etc. are known. Especially, since the MOVPE method can perform uniform film formation to a large area, it is preferred, and the manufacturing method of the 3–5 fellows compound semiconductor of this invention is based on MOVPE method.

[0012]The following raw materials can be used in the manufacturing method of the 3–5 fellows compound semiconductor of this invention. That is, as three group materials, it may be described as trimethylgallium [$(CH_3)_3 Ga$ and the following TMG. It may be described as], triethylgallium [$(C_2H_5)_3 Ga$, and following TEG. general formula $R_1R_2R_3Ga(s)$ (here — R_1 and R_2 .), such as] R_3 shows a low-grade alkyl group. Trialkyl gallium expressed; it may be described as trimethylaluminum [$(CH_3)_3 aluminum$] triethylaluminum [$(C_2H_5)_3 aluminum$ and following TEA.], triisobutylaluminum [$(i-C_4H_9)_3 aluminum$], general formula $R_1R_2R_3aluminum$ (here — R_1 and R_2 .) of ** R_3 shows a low-grade alkyl group. Trialkylaluminium; trimethylamine alane expressed [$(CH_3)_3 N:AlH_3$]; it may be described as trimethylindium [$(CH_3)_3 In$ and the following TMI. The trialkyl indium etc. which are expressed with general formula $R_1R_2R_3In(s)$ (R_1 , R_2 , and R_3 show a low-grade alkyl group here.), such as] and triethylindium [$(C_2H_5)_3 In$], are mentioned. Independent, it mixes and these are used.

[0013]Next, as five group materials, ammonia, hydrazine, methylhydrazine, 1, and 1-dimethylhydrazine, 1, 2-dimethylhydrazine, tert-butylamine, ethylenediamine, etc. are mentioned. Independent, it mixes and these are used. Among these raw materials, since ammonia and hydrazine do not contain a carbon atom in a molecule, contamination of carbon to the inside of a semiconductor is preferred for them few. As a p type dopant of this 3–5 fellows compound semiconductor, two group elements are important. Although Mg, Zn, Cd, Hg, and Be are specifically mentioned, in this, Mg which the p type thing of low resistance tends to build is preferred. As a raw material of Mg dopant, bis(cyclopentadienyl) magnesium, Screw methylcyclopentadienyl magnesium, screw ethylcyclopentadienyl magnesium, General formula $(RC_5H_4)_2 Mg$, such as screw n-propylcyclopentadienyl magnesium and bis-i-propylcyclopentadienyl magnesium. (however, R shows a with H or an or more 1 carbon number [or less 4] low-grade alkyl group.) — since the organic metallic compound expressed has suitable steam pressure, it is suitable. As a n type dopant of this 3–5 fellows compound semiconductor, four group elements and six group elements are important. Although Si, germanium, and O are specifically mentioned, in this, Si from which it is easy to build the n type of low resistance, and what has high raw material purity is obtained is preferred. As a raw material of Si dopant, Silang (SiH_4), a disilane (Si_2H_6), etc. are preferred. Although inactive gas, such as nitrogen and argon, is mentioned as carrier gas, since the thing of a high grade is easy to be obtained, nitrogen is preferred.

[0014]After this invention grows the 1st layer expressed with general formula $In_x Ga_y aluminum_z N$ (however, $x+y+z=1$, $0 < x \leq 1$, $0 \leq y \leq 1$, $0 \leq z \leq 1$), Without supplying three group materials, before carrying out vapor phase

epitaxy of the 2nd layer expressed with general formula In_uGa_v aluminum_wN (however, $u+v+w=1$, $0 \leq u \leq 1$, $0 \leq v \leq 1$, $0 \leq w \leq 1$). It has the process of supplying carrier gas or supplying carrier gas and five group materials. By having the process (it may be hereafter described as a growth interruption process.) of not supplying these three group materials but interrupting growth, Although a reason is not full, when a uniform 3-5 fellows compound semiconductor can be obtained and this 3-5 fellows compound semiconductor is especially used for a light emitting device, the light emitting device which emits light by uniform wavelength and intensity in a substrates face can be obtained. There is an effect prominent about especially the layer that contains InN not less than 5% by a mixed crystal ratio. It is thought by having this growth interruption process that the crystal of the 1st layer is denatured. When growth interruption time is short enough, even if it supplies, it is not necessary to carry out five group materials. However, since the crystallinity of the 1st layer may deteriorate if five group materials are not supplied when growth interruption is long, it is preferred to supply five group materials.

Although it is dependent on temperature, atmosphere, etc. of performing growth interruption, when time to perform growth interruption is too brief, and the effect of growth interruption is not enough and is too long, it is difficult for it to obtain the target InN mixed crystal ratio. As time of desirable growth interruption, 60 or less minutes is mentioned 1 second or more, and it is 30 or less minutes 30 seconds or more still more preferably.

[0015]Hereafter, the effect of a growth interruption process is explained concretely. Drawing 1 is an example of a quantum well structure producible using this compound semiconductor. The buffer layer 2 is grown up to be the substrate 1, and the In_uGa_v aluminum_wN layer 5 which are said In_xGa_y aluminum_zN layer 4 which are the above-mentioned Ga_a aluminum_bN layer 3 and the 1st layer of this invention further, and the 2nd layer of this invention is grown up. . Made the band gap of the Ga_a aluminum_bN layer 3 and the In_uGa_v aluminum_wN layer 5 larger than the In_xGa_y aluminum_zN layer 4. The In_xGa_y aluminum_zN layer 4 turns into a quantum well layer by considering it as what is called double hetero structure, and the strong photoluminescence (it may be hereafter described as PL.) from a quantum well layer is observed. In this case, it mainly depends for the luminous wavelength of PL on the presentation of three group elements of a quantum well layer, and the thickness of a quantum well layer. Strongly reflecting the crystallinity of the structure where PL intensity contains a quantum well layer, PL intensity becomes strong, so that crystallinity is generally high. For this reason, the crystallinity of the laminated structure containing the mixed crystal ratio of a quantum well layer, thickness, and a quantum well and the homogeneity in those substrates faces can be evaluated by evaluating the semiconductor of the structure of drawing 1 using PL. When growth interruption is not performed after growth of the 1st layer but the 2nd layer is grown up immediately, compared with the case where growth interruption is performed, heterogeneity strong against the luminous wavelength and intensity of PL in a substrates face is accepted, and, on the whole, PL intensity is also weak. The luminous wavelength of PL in a substrates face and strong homogeneity improve, and, on the whole, PL intensity also becomes strong as this growth interruption time becomes long. However, since there is a tendency for PL luminous wavelength to become short as growth interruption time becomes long, when performing growth interruption of long time, it is preferred to adjust the mixed crystal ratio of the 1st layer beforehand in consideration of the aforementioned wavelength shift. In the above-mentioned example, although the number of the 1st layer is one, when it is the structure where this two or more layers compound semiconductor was laminated, the laminated structure excellent in homogeneity can be produced by establishing a suitable growth interruption process after growth of each class.

[0016]Next, the light emitting device obtained using the 3-5 fellows compound semiconductor obtained by the manufacturing method of the 3-5 fellows compound semiconductor of this invention is explained. In the quantum well structure shown in drawing 1, it becomes a light emitting device of double hetero structure by giving conductivity which is mutually different in the layer 3 and the layer 5 which touch the luminous layer 4. It is common to use the layer below a luminous layer as a n type from the ease of growth. The semiconductor substrate of a light emitting device with high luminous efficiency excellent in homogeneity can be manufactured by performing growth interruption after growth of a luminous layer. In order to give conductivity to the layer which touches an active layer, an impurity is doped in these layers, but by this doping, the crystallinity of these layers may fall and, as a result, decline in luminous efficiency may be caused. In such a case, luminous efficiency may be able to be improved by providing a layer with low impurity concentration between active layers and these layers. The example of such a structure is shown in drawing 2.

[0017]Although drawing 2 is the example which made the single quantum well layer the luminous layer, the layer which functions as a luminous layer may be a layer which consists of two or more layers. As an example as which the layer which consists of two or more layers concretely functions as a luminous layer, the structure where two or more luminous layers are laminated with the layer with a larger band gap than this is mentioned. When the 1st layer that is a luminous layer contains aluminum, and it is easy to incorporate impurities, such as

O, and uses as a luminous layer, luminous efficiency may fall. In such a case, what is expressed with general formula In_xGa_yN (however, $x+y=1$, $0 < x \leq 1$, $0 \leq y \leq 1$) which does not contain aluminum as a luminous layer can be used.

[0018] As already explained, since it changes with mixed crystal ratios a lot, the grating constant of this 3-5 fellows compound semiconductor must make layer thickness small according to the size of distortion by lattice mismatching, when the grating constant between the layers of this 3-5 fellows compound semiconductor has big lattice mismatching. It depends for the range of desirable thickness on the size of distortion. When an InN mixed crystal ratio laminates this not less than 10% of 3-5 fellows compound semiconductor on said $Ga_aAluminum_bN$, the thickness with a preferred layer containing In is not less than 5A 500A or less. Luminous efficiency becomes less enough when the layer thickness containing In is smaller than 5 A. When larger than 500 A, a defect occurs and luminous efficiency becomes too enough less. The range of still more desirable thickness is not less than 5A 90A or less. By making thickness of a luminous layer small, since an electric charge can be confined in a luminous layer with high density, luminous efficiency can be raised. For this reason, as for the thickness of a luminous layer, even when the difference of a grating constant is smaller than the above-mentioned example, it is preferred to make it be the same as that of the above-mentioned example.

[0019] A luminous layer can be made to emit light on wavelength which is different from the band gap of a luminous layer with doping an impurity. Since this is luminescence from an impurity, it is called impurity luminescence. In impurity luminescence, a luminous wavelength is decided with a presentation and impurity element of three group elements of a luminous layer. In this case, not less than 5% of the InN mixed crystal ratio of a luminous layer is desirable. When an InN mixed crystal ratio is smaller than 5%, most lights which emit light are ultraviolet rays, and cannot sense sufficient luminosity. A luminous wavelength becomes long as In mixed crystal ratio is increased, and a luminous wavelength can be adjusted from purple to blue and green. As an impurity suitable for impurity luminescence, two group elements are preferred. In two group elements, when Mg , Zn , and Cd are doped, since luminous efficiency is high, it is suitable. In particular, Zn is preferred. The concentration of these elements has 10^{18} – preferred $10^{22}cm^{-3}$. The 3rd layer may dope Si or germanium simultaneously with these two group elements. Si and the desirable density range of germanium are $10^{18} – 10^{22}cm^{-3}$.

[0020] In impurity luminescence, an emission spectrum may shift as an emission spectrum generally becomes broadcloth and the amount of injected charges increases. For this reason, it is more advantageous to use band end luminescence, when it is required for the case where high color purity is required, or the narrow wavelength range to centralize emission power. In order to realize the light emitting device by band end luminescence, the quantity of the impurity contained in a luminous layer must be stopped low. Specifically, the concentration of below $10^{19}cm^{-3}$ is [all] preferred about each element of Si , germanium, Mg , Cd , and Zn . It is below $10^{18}cm^{-3}$ still more preferably.

[0021] In band end luminescence, the luminescent color is decided by the presentation of three group elements of a luminous layer. When making light emit by a visible portion, not less than 10% of an InN mixed crystal ratio is desirable. When an InN mixed crystal ratio is smaller than 10%, most lights which emit light are ultraviolet rays, and cannot sense sufficient luminosity. A luminous wavelength becomes long as an InN mixed crystal ratio increases, and a luminous wavelength can be adjusted from purple to blue and green.

[0022] When the mixed crystal ratio of InN in a luminous layer is high, thermal stability is not enough and may cause degradation by the inside of crystal growth, or a semiconductor process. A protection feature can be given to the 2nd layer of this invention which grows up to be the next of a luminous layer for the purpose of preventing degradation of such a luminous layer. In order to give sufficient protection feature for the 2nd layer, not less than 5% of the mixed crystal ratio of AlN is [the mixed crystal ratio of InN of the 2nd layer] desirable 10% or less. Still more preferably, an InN mixed crystal ratio is 5% or less, and an AlN mixed crystal ratio is not less than 10%. As for the thickness of the 2nd layer, in order to give sufficient protection feature for the 2nd layer, not less than 10A 1 micrometer or less is preferred. It is not less than 50A 5000A or less still more preferably. Sufficient effect will not be acquired if the thickness of a protective layer is smaller than 10 A. Since luminous efficiency decreases in being larger than 1 micrometer, it is not desirable.

[0023]

[Example] Hereafter, although this invention is explained still in detail based on an example, this invention is not limited to these.

producing the 3-5 fellows compound semiconductor of the structure shown in drawing 1 with example 1 MOVPE method, and measuring the spectrum of PL -- the field of a luminescent state -- internal division -- cloth was evaluated. Organic washing was carried out and what carried out mirror polishing of the sapphire C side (25 mm x

25 mm) as a substrate was used. Growth used the two-step growth method which uses GaN as a low-temperature growth buffer layer. GaN layer [in ordinary pressure] 3 about 2.5 micrometers thick was grown up. Next, 0.5 atmosphere and substrate temperature shall be 785 **, use carrier gas as nitrogen for a reactor pressure, and 4slm supply of carrier gas, TEG, TMI, and the ammonia is carried out 0.4 sccm 0.04 sccm 6 slm, respectively, The $In_{0.3}Ga_{0.7}N$ layer 4 which is the 1st layer of this invention was grown up for 70 seconds.

However, slm and sccm show that the gas of the weight in which 1slm occupies per minute in the unit of a gaseous flow, and occupies a volume of 1 l. by a normal condition is flowing, and 1000sccm is equivalent to 1slm. [0024]After holding the state of supplying only nitrogen and ammonia as a growth interruption process, for 5 minutes, 4slm supply of TEG, TEA, and the ammonia is carried out 0.008 sccm 0.032 sccm at the still more nearly same temperature, respectively, The $Ga_{0.8}aluminum_{0.2}N$ layer 5 which is the 2nd layer of this invention

was grown up for 10 minutes. Since the growth rate for which it asked on the same conditions about the thickness of this layer 4 and the layer 5 from the still longer layer thickness which carried out time growth is a part for part 30A/for 43A/, respectively, the thickness called for from the above-mentioned growth time is calculable with 50 A and 300 A, respectively. When 325-nm luminescence of helium-Cd laser was made into the excitation light source for the 3-5 follows compound semiconductor sample produced by the above and PL measurement at a room temperature was performed, luminescence whose peak wavelength of near 5000A is strong was accepted the whole surface within the substrates face except 5 mm of circumferences. Typical PL spectrum is shown in drawing 3. The output of the detector in the peak wavelength of a spectrum was 7.4 mV. [0025]Except for the time of example 2 growth interruption process being 2 minutes, the sample was produced like Example 1. When PL measurement at the room temperature of this sample was performed like Example 1, the weak portion of PL was also selectively accepted in within the substrates face except 5 mm of circumferences, but most showed strong PL like Example 1. It is shown in typical PL spectrum drawing 4 of the strong part of PL, and a weak portion. The surface ratio occupied in the substrates face of the strong portion of PL and a weak portion was 3:1 about. The peak intensity of the spectrum from the strong portion of PL and a weak portion was 4 mV and 0.17 mV about, respectively.

[0026]Except for not having performed growth interruption but having grown up the $Ga_{0.8}aluminum_{0.2}N$ layer 5, the sample was produced like Example 1 after growth of the comparative example 1 1st layer 4. When PL in the room temperature was evaluated like Example 1 about this sample, as for the portion which emits light, luminescence was not accepted almost over the whole surface of a certain thing. PL spectrum of the portion which emits light most strongly is shown in drawing 5. The peak intensity was only about 0.1 mV.

[0027]Form GaN buffer layer 2 like example 3 Example 1, and TMG, ammonia, and silane gas are supplied at 1100 **, GaN layer 6 of 2.5 micrometers of n type thickness which doped Si is grown up, TMG and ammonia are supplied at the still more nearly same temperature, and 1500A grows non-doped GaN layer 7. Next, substrate temperature is lowered to 785 **, TEG, TMI, and ammonia are supplied by making nitrogen into carrier gas, and 50A grows the $In_{0.3}Ga_{0.7}N$ layer 4. After holding the state of supplying only nitrogen and ammonia, for 5 minutes, TEG, TEA, and ammonia are supplied at the still more nearly same temperature, and 300A grows the $Ga_{0.8}aluminum_{0.2}N$ layer 5. Next, substrate temperature is raised to 1100 ** and 5000A grows GaN layer 8 which supplied TMG, Cp2Mg, and ammonia and doped Mg. After taking out the 3-5 follows compound semiconductor sample produced by the above from a reactor, annealing treatment is performed in nitrogen and the GaN layer which doped Mg is made into the p type layer of low resistance. In this way, an electrode can be formed in the obtained sample with a conventional method, and LED can be produced with sufficient homogeneity in a substrates face by being referred to as LED.

[0028]

[Effect of the Invention]It is uniform, according to this invention, when the high 3-5 follows compound semiconductor thin film of the yield can be manufactured and it uses especially as a light emitting device, a luminescent state is uniform, the high light emitting device of the yield can be obtained, it is very useful, and industrial value is large.

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TECHNICAL FIELD

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PRIOR ART

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[0003] Since a crystal with this compound semiconductor good in bulk growth is not obtained, homoepitaxial growth using this compound semiconductor itself as a substrate is difficult. By the way, it is known that a good crystalline thing can be obtained because the compound semiconductor expressed with general formula Ga_aAlN (however, $a+b=1$, $0 \leq a \leq 1$, $0 \leq b \leq 1$) uses buffer layers, such as GaN and AlN. This Ga_aAlN has the five same group elements as this In_xGa_yAlN , and since a crystal structure is also the same, this In_xGa_yAlN can obtain a high crystalline thing by growing up on the above-mentioned Ga_aAlN .

[0004] By the way, the grating constant of this compound semiconductor changes with mixed crystal ratios a lot. Especially, to GaN or AlN, about 12% or more, since it is large, depending on the mixed crystal ratio of each class of this compound semiconductor, a big difference may produce the grating constant of InN in the grating constant between layers. When there is big lattice mismatching, a defect may arise into a crystal and it becomes the cause of reducing crystallinity. It is difficult to realize high luminous efficiency in the light emitting device which generally produced the defect using the crystal included mostly.

[0005] In order to suppress generating of the defect by lattice mismatching, according to the size of distortion by lattice mismatching, layer thickness must be made small. However, when thickness made a film an active layer dramatically, it was difficult for the physical properties of a luminous layer to receive influence also in the mixed crystal ratio of a luminous layer or the deflection of some of thickness, and to produce the target luminous wavelength or the light emitting device of luminescence intensity uniformly covering all the substrates faces.

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EFFECT OF THE INVENTION

[Effect of the Invention]It is uniform, according to this invention, when the high 3–5 fellows compound semiconductor thin film of the yield can be manufactured and it uses especially as a light emitting device, a luminescent state is uniform, the high light emitting device of the yield can be obtained, it is very useful, and industrial value is large.

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention]It is uniform, the purpose of this invention has a uniform luminescent state, when it uses as the manufacturing method of the 3-5 fellows compound semiconductor which can manufacture the high 3-5 fellows compound semiconductor thin film of the yield, especially a light emitting device, and there is in providing the manufacturing method of the 3-5 fellows compound semiconductor for light emitting devices which can obtain the high light emitting device of the yield.

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MEANS

[Means for Solving the Problem] This invention persons found out that a uniform thin film was obtained for high quality by Lycium chinense in a process of stopping supply of three group materials as a result of wholeheartedly examination by the time it grows up the following layer after growing up a thin film of this compound semiconductor about a growing condition of a thin film of this 3-5 fellows compound semiconductor, and resulted in this invention.

[0008] That is, this invention is an invention described below.

[1] Vapor phase epitaxy of the 1st layer of a 3-5 fellows compound semiconductor expressed with general formula In_xGa_yAlN (however, $x+y+z=1$, $0 < x \leq 1$, $0 \leq y \leq 1$, $0 \leq z \leq 1$) is carried out, Next, in a method of carrying out vapor phase epitaxy of the 2nd layer expressed with general formula In_uGa_vAlN (however, $u+v+w=1$, $0 \leq u \leq 1$, $0 \leq v \leq 1$, $0 \leq w \leq 1$), and manufacturing a 3-5 fellows compound semiconductor, A manufacturing method of a 3-5 fellows compound semiconductor having the process of supplying carrier gas or supplying carrier gas and five group materials without supplying three group materials before growth of the 2nd layer, after growing up the 1st layer.

[2] It is characterized by a mixed crystal ratio of InN of the 1st layer being not less than 5%. [1] A manufacturing method of a 3-5 fellows compound semiconductor of a statement.

[0009][3] Thickness of the 1st layer is characterized by not less than 10A being 500A or less. [1] or [2] A manufacturing method of a 3-5 fellows compound semiconductor of a statement.

[4] Each concentration of each element of Si, germanium, Cd and Zn which are contained in the 1st layer, and Mg is characterized by being below $10^{19}cm^{-3}$. [1]**[2] or [3] A manufacturing method of a 3-5 fellows compound semiconductor of a statement.

[0010]

[Embodiment of the Invention] Next, this invention is explained in detail. Generally as a substrate for crystal growth of this 3-5 fellows compound semiconductor, sapphire, ZnO , $GaAs$, Si , SiC , NGO ($NdGaO_3$), a spinel ($MgAl_2O_4$), etc. are used. Since the quality crystal of a large area is obtained transparently, especially sapphire is preferred.

[0011] as the manufacturing method of this 3-5 fellows compound semiconductor -- molecular beam epitaxy (it may be hereafter described as MBE.) -- law and organic metal vapor growth (it may be hereafter described as MOVPE.) -- law and hydride vapor phase epitaxy (it may be hereafter described as HVPE.) -- law etc. are known. Especially, since the MOVPE method can perform uniform film formation to a large area, it is preferred, and the manufacturing method of the 3-5 fellows compound semiconductor of this invention is based on MOVPE method.

[0012] The following raw materials can be used in the manufacturing method of the 3-5 fellows compound semiconductor of this invention. That is, as three group materials, it may be described as trimethylgallium [$(CH_3)_3Ga$ and the following TMG. It may be described as], triethylgallium [$(C_2H_5)_3Ga$, and following TEG. general formula $R_1R_2R_3Ga(s)$ (here -- R_1 and R_2 .), such as] R_3 shows a low-grade alkyl group. Trialkyl gallium expressed; it may be described as trimethylaluminum [$(CH_3)_3Al$ aluminum] triethylaluminum [$(C_2H_5)_3Al$ aluminum and following TEA.], triisobutylaluminum [$(i-C_4H_9)_3Al$ aluminum], general formula $R_1R_2R_3Al$ aluminum (here -- R_1 and R_2 .) of ** R_3 shows a low-grade alkyl group. Trialkylaluminium; trimethylamine alane expressed [$(CH_3)_3N:AlH_3$]; it may be described as trimethylindium [$(CH_3)_3In$ and the following TMI. The trialkyl indium etc. which are expressed with general formula $R_1R_2R_3In(s)$ (R_1 , R_2 , and R_3 show a low-grade alkyl group here.), such as] and triethylindium [$(C_2H_5)_3In$], are mentioned. Independent, it mixes and these are used.

[0013] Next, as five group materials, ammonia, hydrazine, methylhydrazine, 1, and 1-dimethylhydrazine, 1, 2-dimethylhydrazine, tert-butylamine, ethylenediamine, etc. are mentioned. Independent, it mixes and these are used. Among these raw materials, since ammonia and hydrazine do not contain a carbon atom in a molecule, contamination of carbon to the inside of a semiconductor is preferred for them few. As a p type dopant of this 3-5 fellows compound semiconductor, two group elements are important. Although Mg, Zn, Cd, Hg, and Be are specifically mentioned, in this, Mg which the p type thing of low resistance tends to build is preferred. As a raw material of Mg dopant, bis(cyclopentadienyl) magnesium, Screw methylcyclopentadienyl magnesium, screw ethylcyclopentadienyl magnesium, General formula $(RC_5H_4)_2Mg$, such as screw n-propylcyclopentadienyl magnesium and bis-i-propylcyclopentadienyl magnesium. (however, R shows a with H or an or more 1 carbon number [or less 4] low-grade alkyl group.) -- since the organic metallic compound expressed has suitable steam pressure, it is suitable. As a n type dopant of this 3-5 fellows compound semiconductor, four group elements and six group elements are important. Although Si, germanium, and O are specifically mentioned, in this, Si from which it is easy to build the n type of low resistance, and what has high raw material purity is obtained is preferred. As a raw material of Si dopant, Silang (SiH_4), a disilane (Si_2H_6), etc. are preferred. Although inactive gas, such as nitrogen and argon, is mentioned as carrier gas, since the thing of a high grade is easy to be obtained, nitrogen is preferred.

[0014] After this invention grows the 1st layer expressed with general formula In_xGa_y aluminum_zN (however, $x+y+z=1$, $0 < x \leq 1$, $0 \leq y \leq 1$, $0 \leq z \leq 1$), Without supplying three group materials, before carrying out vapor phase epitaxy of the 2nd layer expressed with general formula In_uGa_v aluminum_wN (however, $u+v+w=1$, $0 \leq u \leq 1$, $0 \leq v \leq 1$, $0 \leq w \leq 1$), It has the process of supplying carrier gas or supplying carrier gas and five group materials. By having the process (it may be hereafter described as a growth interruption process.) of not supplying these three group materials but interrupting growth, Although a reason is not full, when a uniform 3-5 fellows compound semiconductor can be obtained and this 3-5 fellows compound semiconductor is especially used for a light emitting device, the light emitting device which emits light by uniform wavelength and intensity in a substrates face can be obtained. There is an effect prominent about especially the layer that contains InN not less than 5% by a mixed crystal ratio. It is thought by having this growth interruption process that the crystal of the 1st layer is denatured. When growth interruption time is short enough, even if it supplies, it is not necessary to carry out five group materials. However, since the crystallinity of the 1st layer may deteriorate if five group materials are not supplied when growth interruption is long, it is preferred to supply five group materials.

Although it is dependent on temperature, atmosphere, etc. of performing growth interruption, when time to perform growth interruption is too brief, and the effect of growth interruption is not enough and is too long, it is difficult for it to obtain the target InN mixed crystal ratio. As time of desirable growth interruption, 60 or less minutes is mentioned 1 second or more, and it is 30 or less minutes 30 seconds or more still more preferably.

[0015] Hereafter, the effect of a growth interruption process is explained concretely. Drawing 1 is an example of a quantum well structure producible using this compound semiconductor. The buffer layer 2 is grown up to be the substrate 1, and the In_uGa_v aluminum_wN layer 5 which are said In_xGa_y aluminum_zN layer 4 which are the above-mentioned Ga_a aluminum_bN layer 3 and the 1st layer of this invention further, and the 2nd layer of this invention is grown up. . Made the band gap of the Ga_a aluminum_bN layer 3 and the In_uGa_v aluminum_wN layer 5 larger than the In_xGa_y aluminum_zN layer 4. The In_xGa_y aluminum_zN layer 4 turns into a quantum well layer by considering it as what is called double hetero structure, and the strong photoluminescence (it may be hereafter described as PL.) from a quantum well layer is observed. In this case, it mainly depends for the luminous wavelength of PL on the presentation of three group elements of a quantum well layer, and the thickness of a quantum well layer. Strongly reflecting the crystallinity of the structure where PL intensity contains a quantum well layer, PL intensity becomes strong, so that crystallinity is generally high. For this reason, the crystallinity of the laminated structure containing the mixed crystal ratio of a quantum well layer, thickness, and a quantum well and the homogeneity in those substrates faces can be evaluated by evaluating the semiconductor of the structure of drawing 1 using PL. When growth interruption is not performed after growth of the 1st layer but the 2nd layer is grown up immediately, compared with the case where growth interruption is performed, heterogeneity strong against the luminous wavelength and intensity of PL in a substrates face is accepted, and, on the whole, PL intensity is also weak. The luminous wavelength of PL in a substrates face and strong homogeneity improve, and, on the whole, PL intensity also becomes strong as this growth interruption time becomes long. However, since there is a tendency for PL luminous wavelength to become short as growth interruption time becomes long, when performing growth interruption of long time, it is preferred to adjust the mixed crystal ratio of the 1st layer beforehand in consideration of the aforementioned wavelength shift. In the

above-mentioned example, although the number of the 1st layer is one, when it is the structure where this two or more layers compound semiconductor was laminated, the laminated structure excellent in homogeneity can be produced by establishing a suitable growth interruption process after growth of each class.

[0016]Next, the light emitting device obtained using the 3–5 fellows compound semiconductor obtained by the manufacturing method of the 3–5 fellows compound semiconductor of this invention is explained. In the quantum well structure shown in drawing 1, it becomes a light emitting device of double hetero structure by giving conductivity which is mutually different in the layer 3 and the layer 5 which touch the luminous layer 4. It is common to use the layer below a luminous layer as a n type from the ease of growth. The semiconductor substrate of a light emitting device with high luminous efficiency excellent in homogeneity can be manufactured by performing growth interruption after growth of a luminous layer. In order to give conductivity to the layer which touches an active layer, an impurity is doped in these layers, but by this doping, the crystallinity of these layers may fall and, as a result, decline in luminous efficiency may be caused. In such a case, luminous efficiency may be able to be improved by providing a layer with low impurity concentration between active layers and these layers. The example of such a structure is shown in drawing 2.

[0017]Although drawing 2 is the example which made the single quantum well layer the luminous layer, the layer which functions as a luminous layer may be a layer which consists of two or more layers. As an example as which the layer which consists of two or more layers concretely functions as a luminous layer, the structure where two or more luminous layers are laminated with the layer with a larger band gap than this is mentioned. When the 1st layer that is a luminous layer contains aluminum, and it is easy to incorporate impurities, such as O, and uses as a luminous layer, luminous efficiency may fall. In such a case, what is expressed with general formula In_xGa_yN (however, $x+y=1$, $0 < x \leq 1$, $0 \leq y \leq 1$) which does not contain aluminum as a luminous layer can be used.

[0018]As already explained, since it changes with mixed crystal ratios a lot, the grating constant of this 3–5 fellows compound semiconductor must make layer thickness small according to the size of distortion by lattice mismatching, when the grating constant between the layers of this 3–5 fellows compound semiconductor has big lattice mismatching. It depends for the range of desirable thickness on the size of distortion. When an InN mixed crystal ratio laminates this not less than 10% of 3–5 fellows compound semiconductor on said $Ga_aAluminum_bN$, the thickness with a preferred layer containing In is not less than 5A 500A or less. Luminous efficiency becomes less enough when the layer thickness containing In is smaller than 5 A. When larger than 500 A, a defect occurs and luminous efficiency becomes too enough less. The range of still more desirable thickness is not less than 5A 90A or less. By making thickness of a luminous layer small, since an electric charge can be confined in a luminous layer with high density, luminous efficiency can be raised. For this reason, as for the thickness of a luminous layer, even when the difference of a grating constant is smaller than the above-mentioned example, it is preferred to make it be the same as that of the above-mentioned example.

[0019]A luminous layer can be made to emit light on wavelength which is different from the band gap of a luminous layer with doping an impurity. Since this is luminescence from an impurity, it is called impurity luminescence. In impurity luminescence, a luminous wavelength is decided with a presentation and impurity element of three group elements of a luminous layer. In this case, not less than 5% of the InN mixed crystal ratio of a luminous layer is desirable. When an InN mixed crystal ratio is smaller than 5%, most lights which emit light are ultraviolet rays, and cannot sense sufficient luminosity. A luminous wavelength becomes long as In mixed crystal ratio is increased, and a luminous wavelength can be adjusted from purple to blue and green. As an impurity suitable for impurity luminescence, two group elements are preferred. In two group elements, when Mg, Zn, and Cd are doped, since luminous efficiency is high, it is suitable. In particular, Zn is preferred. The concentration of these elements has 10^{18} – preferred $10^{22}cm^{-3}$. The 3rd layer may dope Si or germanium simultaneously with these two group elements. Si and the desirable density range of germanium are 10^{18} – $10^{22}cm^{-3}$.

[0020]In impurity luminescence, an emission spectrum may shift as an emission spectrum generally becomes broadcloth and the amount of injected charges increases. For this reason, it is more advantageous to use band end luminescence, when it is required for the case where high color purity is required, or the narrow wavelength range to centralize emission power. In order to realize the light emitting device by band end luminescence, the quantity of the impurity contained in a luminous layer must be stopped low. Specifically, the concentration of below $10^{19}cm^{-3}$ is [all] preferred about each element of Si, germanium, Mg, Cd, and Zn. It is below $10^{18}cm^{-3}$ still more preferably.

[0021]In band end luminescence, the luminescent color is decided by the presentation of three group elements of a luminous layer. When making light emit by a visible portion, not less than 10% of an InN mixed crystal ratio is

desirable. When an InN mixed crystal ratio is smaller than 10%, most lights which emit light are ultraviolet rays, and cannot sense sufficient luminosity. A luminous wavelength becomes long as an InN mixed crystal ratio increases, and a luminous wavelength can be adjusted from purple to blue and green.

[0022]When the mixed crystal ratio of InN in a luminous layer is high, thermal stability is not enough and may cause degradation by the inside of crystal growth, or a semiconductor process. A protection feature can be given to the 2nd layer of this invention which grows up to be the next of a luminous layer for the purpose of preventing degradation of such a luminous layer. In order to give sufficient protection feature for the 2nd layer, not less than 5% of the mixed crystal ratio of AlN is [the mixed crystal ratio of InN of the 2nd layer] desirable 10% or less. Still more preferably, an InN mixed crystal ratio is 5% or less, and an AlN mixed crystal ratio is not less than 10%. As for the thickness of the 2nd layer, in order to give sufficient protection feature for the 2nd layer, not less than 10A 1 micrometer or less is preferred. It is not less than 50A 5000A or less still more preferably. Sufficient effect will not be acquired if the thickness of a protective layer is smaller than 10 A. Since luminous efficiency decreases in being larger than 1 micrometer, it is not desirable.

[Translation done.]

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EXAMPLE

[Example]Hereafter, although this invention is explained still in detail based on an example, this invention is not limited to these.

producing the 3-5 fellows compound semiconductor of the structure shown in drawing 1 with example 1 MOVPE method, and measuring the spectrum of PL -- the field of a luminescent state -- internal division -- cloth was evaluated. Organic washing was carried out and what carried out mirror polishing of the sapphire C side (25 mm x 25 mm) as a substrate was used. Growth used the two-step grown method which uses GaN as a low-temperature growth buffer layer. GaN layer [in ordinary pressure] 3 about 2.5 micrometers thick was grown up. Next, 0.5 atmosphere and substrate temperature shall be 785 **, use carrier gas as nitrogen for a reactor pressure, and 4slm supply of carrier gas, TEG, TMI, and the ammonia is carried out 0.4 sccm 0.04 sccm 6 slm, respectively, The $In_{0.3}Ga_{0.7}N$ layer 4 which is the 1st layer of this invention was grown up for 70 seconds.

However, slm and sccm show that the gas of the weight in which 1slm occupies per minute in the unit of a gaseous flow, and occupies a volume of 1 l. by a normal condition is flowing, and 1000sccm is equivalent to 1slm.

[0024]After holding the state of supplying only nitrogen and ammonia as a growth interruption process, for 5 minutes, 4slm supply of TEG, TEA, and the ammonia is carried out 0.008 sccm 0.032 sccm at the still more nearly same temperature, respectively, The $Ga_{0.8}aluminum_{0.2}N$ layer 5 which is the 2nd layer of this invention

was grown up for 10 minutes. Since the growth rate for which it asked on the same conditions about the thickness of this layer 4 and the layer 5 from the still longer layer thickness which carried out time growth is a part for part 30A/for 43A/, respectively, the thickness called for from the above-mentioned growth time is calculable with 50 A and 300 A, respectively. When 325-nm luminescence of helium-Cd laser was made into the excitation light source for the 3-5 fellows compound semiconductor sample produced by the above and PL measurement at a room temperature was performed, luminescence whose peak wavelength of near 5000A is strong was accepted the whole surface within the substrates face except 5 mm of circumferences. Typical PL spectrum is shown in drawing 3. The output of the detector in the peak wavelength of a spectrum was 7.4 mV.

[0025]Except for the time of example 2 growth interruption process being 2 minutes, the sample was produced like Example 1. When PL measurement at the room temperature of this sample was performed like Example 1, the weak portion of PL was also selectively accepted in within the substrates face except 5 mm of circumferences, but most showed strong PL like Example 1. It is shown in typical PL spectrum drawing 4 of the strong part of PL, and a weak portion. The surface ratio occupied in the substrates face of the strong portion of PL and a weak portion was 3:1 about. The peak intensity of the spectrum from the strong portion of PL and a weak portion was 4 mV and 0.17 mV about, respectively.

[0026]Except for not having performed growth interruption but having grown up the $Ga_{0.8}aluminum_{0.2}N$ layer 5, the sample was produced like Example 1 after growth of the comparative example 1 1st layer 4. When PL in the room temperature was evaluated like Example 1 about this sample, as for the portion which emits light, luminescence was not accepted almost over the whole surface of a certain thing. PL spectrum of the portion which emits light most strongly is shown in drawing 5. The peak intensity was only about 0.1 mV.

[0027]Form GaN buffer layer 2 like example 3 Example 1, and TMG, ammonia, and silane gas are supplied at 1100 **, GaN layer 6 of 2.5 micrometers of n type thickness which doped Si is grown up, TMG and ammonia are supplied at the still more nearly same temperature, and 1500A grows non-doped GaN layer 7. Next, substrate temperature is lowered to 785 **, TEG, TMI, and ammonia are supplied by making nitrogen into carrier gas, and 50A grows the $In_{0.3}Ga_{0.7}N$ layer 4. After holding the state of supplying only nitrogen and ammonia, for 5 minutes, TEG, TEA, and ammonia are supplied at the still more nearly same temperature, and 300A grows the $Ga_{0.8}aluminum_{0.2}N$ layer 5. Next, substrate temperature is raised to 1100 ** and 5000A grows GaN layer 8 which supplied TMG, Cp2Mg, and ammonia and doped Mg. After taking out the 3-5 fellows compound semiconductor

sample produced by the above from a reactor, annealing treatment is performed in nitrogen and the GaN layer which doped Mg is made into the p type layer of low resistance. In this way, an electrode can be formed in the obtained sample with a conventional method, and LED can be produced with sufficient homogeneity in a substrates face by being referred to as LED.

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1]The figure showing the structure of the 3-5 fellows compound semiconductor in connection with Example 1.

[Drawing 2]The figure showing the structure of the compound semiconductor light emitting element in connection with Example 3.

[Drawing 3]The figure showing PL spectrum in Example 1.

[Drawing 4]The figure showing PL spectrum in Example 2.

[Drawing 5]The figure showing PL spectrum in the comparative example 1.

[Description of Notations]

- 1 -- Substrate
- 2 -- Buffer layer
- 3 -- $\text{Ga}_a\text{aluminum}_b\text{N}$ layer
- 4 -- $\text{In}_x\text{Ga}_y\text{aluminum}_z\text{N}$ layer
- 5 -- $\text{In}_u\text{Ga}_v\text{aluminum}_w\text{N}$ layer
- 6 -- N type GaN layer
- 7 -- Non-doped GaN layer
- 8 -- P type GaN layer

[Translation done.]

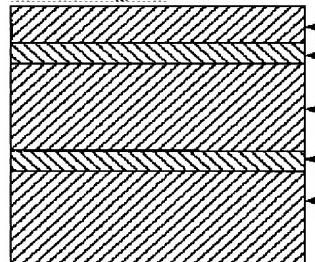
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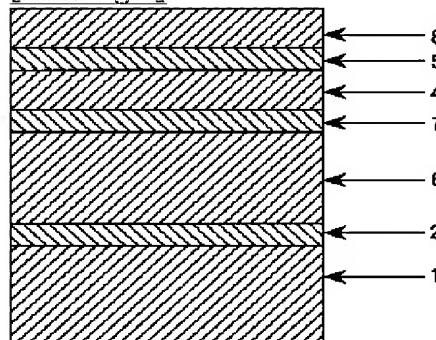
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DRAWINGS

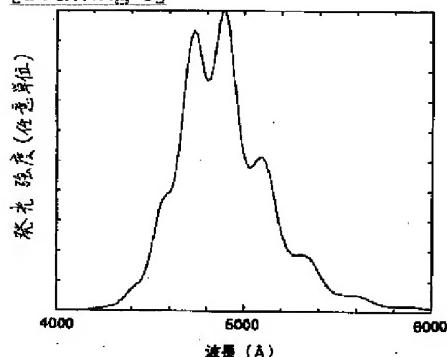
[Drawing 1]



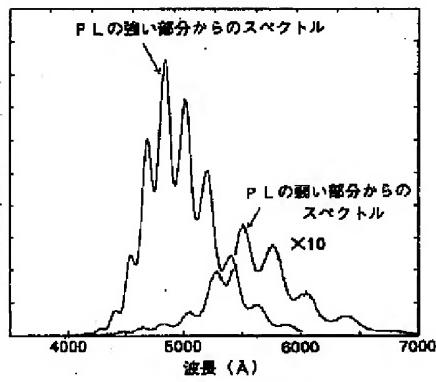
[Drawing 2]



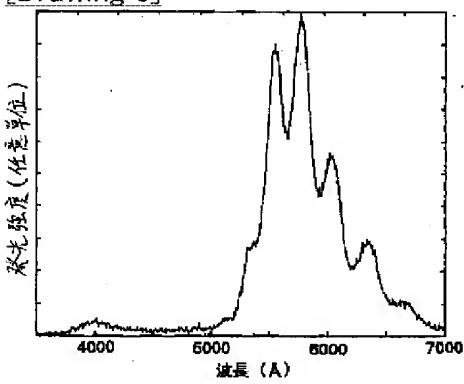
[Drawing 3]



[Drawing 4]



[Drawing 5]



[Translation done.]